

12

EUROPEAN PATENT APPLICATION

21 Application number: 90300809.2

51 Int. Cl.⁵: A61C 13/087, A61K 6/08

22 Date of filing: 25.01.90

30 Priority: 25.01.89 JP 14011/89
 28.02.89 JP 45133/89

43 Date of publication of application:
 01.08.90 Bulletin 90/31

84 Designated Contracting States:
 DE GB

71 Applicant: JAPAN INSTITUTE OF ADVANCED
 DENTISTRY
 2-1-47, Kanda Surugadai Chiyoda-ku.
 Tokyo 101(JP)

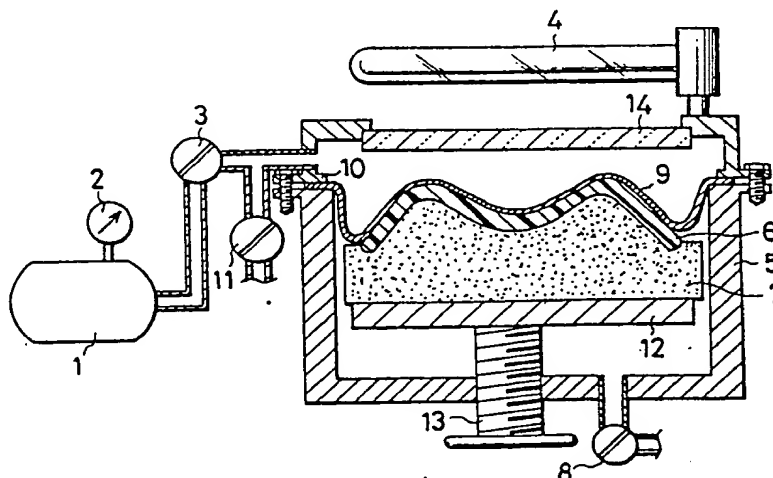
72 Inventor: Masuhara, Eiichi
 2-5-10 Honkomagome, Bunkyo-ku
 Tokyo(JP)
 Inventor: Komiya, Shigeo
 3-7-10 Minami-Urawa
 Urawa-shi, Saitama-ken(JP)
 Inventor: Makino, Shin
 2-13-8 Futamata
 Ichikawa-shi, Chiba-ken(JP)

74 Representative: Senior, Alan Murray et al
 J.A. KEMP & CO 14 South Square Gray's Inn
 London WC1R 5EU(GB)

54 A method for light curing of dental light-curing resins and an apparatus therefor.

57 The present invention provides a method for light curing of dental light-curing resins, characterized in that gas pressure is exerted on the resins during the light-curing, and a apparatus for use in the light curing. According to the present method, the resulting cured resins have no bubble therein. They are dense and their surfaces are smooth, shiny and very aesthetic. Furthermore, their bending strength is very high.

Fig. 1



EP 0 380 345 A1

A METHOD FOR LIGHT CURING OF DENTAL LIGHT-CURING RESINS AND AN APPARATUS THEREFOR

The present invention relates to a method and apparatus for light curing of dental light-curing resins so as to improve the properties of the resultant cured resins, such as surface finish.

In recent years, light-curing resins have been employed in the preparation of dental laboratory products (prostheses) for use in dentures, removable space maintainers, removable orthodontic appliances, crowns and bridges, and the like.

Light-curing resins are easier to handle and faster to polymerize than conventional self-curing resins or heat-curing resins which require powder and solution to be mixed with each other. They have a further advantage in that the resulting products include only a few air bubbles and exhibit good properties since they have been prepared in a one-paste form in advance.

However, conventional light-curing denture base resins are treated in such a manner that the resin paste is finger-pressed and fixed onto a stone cast and then light-cured. This process is time-consuming and requires skill in order to obtain an air bubble-free surface with uniform thickness.

In addition, although the air bubbles within the resin paste are invisible by the naked eye, minute air bubbles have often been apparent on a surface in contact with the stone cast when light-cured under normal pressure, because the resin paste detaches from the surface of the stone cast through polymerization and contraction.

Accordingly, the resin prosthesis thus has a poor surface finish and its mechanical resistance is unsatisfactory.

Moreover, in the process of light curing of crown and bridge-veneering resins wherein the resin paste is built up, using an instrument, on an opaque resin surface applied to the metal surface of facing, and then light-cured under normal pressure, contraction takes place at a laminated part between the opaque resin surface and the resin paste before the completion of the light curing of crown and bridge-veneering resins. As a result, satisfactory adhesion can not be obtained.

When the resulting prosthesis is used, the laminated part is liable to break inside the mouth. Application of an adhesive primer, for example, has been tried for preventing such failure, but this has proven to be an unsatisfactory solution.

Furthermore, conventional light-curing resins tend to leave a greater unpolymerized layer than heat-curing resins because they are polymerized and cured at room temperature. Thus, the cured product has poor mechanical resistance, and absorbs much water. Accordingly, it is highly soluble and lacks long-term durability inside the mouth.

According to a first aspect of the present invention there is provided a method for light curing of dental light-curing resins, characterised in that gas pressure above ambient pressure is exerted on the resin during the light-curing.

The surface of the dental light-curing resins are preferably covered by a transparent or translucent, flexible sheet and the gas pressure is exerted through the sheet.

Preferably, the dental light-curing resins are pressed and fixed on a support and the sheet is heated before or during the light curing.

According to a second aspect of the present invention there is provided an apparatus for light curing of resins comprising: a chamber, lighting means and means for selectively exerting a gas pressure above ambient pressure on the resin in the chamber.

In order that the invention can be better understood, the following description is given with reference to the accompanying drawings, given by way of example only and in which:-

Figure 1 shows a partial cross-section of the apparatus which is used in Examples 1, 2 and 3 of the description.

Figure 2 shows a partial cross-section of the apparatus which is used in Example 4 of the description.

Figure 3 shows a partial cross-section of the apparatus which is used in Example 5 of the description.

Gas pressure may be exerted, for example, by means of compressed gas provided by a compressor or gas cylinder connected to the light-curing unit. The pressure and kind of gas within the unit may be optionally selected according to purpose or application. When it is necessary to keep an unpolymerized layer on the surface of the light-cured resins for successively laminating light-curing resins, air or oxygen is preferably used as the compressed gas. On the other hand, if complete polymerization of the resin is desired, an inert gas such as nitrogen, argon or helium gas is preferably used. The range of gas pressures within the light-curing unit is not fixed, but generally it is from 1 to 20 bar, preferably from 2 to 10 bar. Pressure lower than 1 bar can not effectively press the resin surface, and pressure greater than 20 bar may disadvantageously press the prosthesis to be too thin. The preferred pressure range is from 2 to 7 bar.

When one presses and fixes the dental light-curing resins on the support, finger pressure is normally employed in order to evenly press on the upper surface of the resins. The support may, for example, be a stone cast, a resin cast or a denture base.

The transparent or translucent, flexible sheet may be of polyethylene, polyethylene-vinyl acetate copolymer, polyvinyl chloride, polyvinylidene chloride, polyvinylidene fluoride, polyurethane or polysiloxane. Among them, polyvinyl chloride, polyethylene-vinyl acetate copolymer, urethane or silicon rubber are especially preferable, since they allow repeated pressing and fixing onto the light-curing resins without wrinkling.

Apart from polyurethane and polysiloxane, it is preferable to heat the sheet so that it is more flexible. As a heating unit of the present invention, an electrical resistance heater, infrared-ray heater, far infrared-ray heater, or microwave may be used. The heater may be positioned depending upon the type of dental prosthesis. The temperature and heating time may be optionally selected, being usually around 200°C and for a few minutes. The heating of the sheet may be performed before or during the light curing but preferably before setting the sheet in the light curing unit.

In order to heat the sheet for adaptation, it is also preferable that the compressed gas should be heated to a temperature in the range 80°C to 250°C as it is supplied. Therefore, it is recommended to position a heater (e.g. an electrical heater) along a gas conduit and before a valve for conducting the compressed gas.

The transparent or translucent and flexible sheet is very useful for uniformly transmitting the exerted gas pressure to all parts of the dental light-curing resin.

The sheet is especially useful when it has to be pressfixed on a wide range of surfaces, e.g. denture bases or removable orthodontic appliances.

When the light-curing resins are light-cured while being subjected to gas-pressure through the sheet, no deformation such as curvature due to contraction by the light-curing will occur because the resins are pressed by the sheet at all times during the process. Also, the light curing process will not be interrupted since the resin surface does not come in a direct contact with the gas, e.g. air.

A part for fastening the transparent or translucent and flexible sheet may be fitted in the apparatus so as to seal and fasten the edge of the sheet placed inside the light-curing unit.

As a lighting unit, a lamp is used which preferably generates visible light, such as a xenon lamp, halogen lamp or fluorescent lamp with a filter, if necessary, for filtering out ultraviolet and infrared rays.

Although the lamp may be placed either inside or outside the light-curing unit, it is preferable to place it on the outside, considering the high gas pressure in the unit and durability of the lamp.

The degree of the light curing of the present invention may be controlled by adjusting the distance between the light-curing resin and the light source (e.g. halogen lamp). For this purpose, either the light source or the resin may be arranged to be movable.

It is thus desirable that a part of the light-curing unit is made of transparent material so that the resins in the unit can be irradiated through the transparent material from the outside.

The nature of exemplary embodiments of apparatus according to the invention will become apparent from the following Examples which describe particular ways in which these embodiments may be employed.

EXAMPLE 1 - see Figure 1.

At first, a resin sheet of EPOREX-D (light-curing denture base resin by Nippon Oil and Fats Co. Ltd) 6 was placed on top of a stone cast 7 having the shape of an edentulous jaw, evenly finger-pressed and then set on a bed plate 12.

Then a flexible silicon rubber (polysiloxane) sheet 9 was placed on a sheet fastener 10 and fixed, and after a screw height adjuster 13 was adjusted to control the position of the stone cast 7, a first air valve 8 and a second air valve 11 were closed.

Thereafter, by opening an air valve 3, compressed air in an air compressor 1 with a pressure gauge 2 was allowed to flow into a light-curing unit 5, which was provided with a quartz glass board 14, until the gas pressure in the unit reached 5 bar. The quartz glass board may be coated with a filter agent for filtering out infrared rays.

After closing the valve 3, the resin sheet 6 was light-cured for five minutes using 27W U-LINE 2 (a fluorescent lamp manufactured by TOSHIBA CORPORATION) 4 which emitted visible light with a wave length of 400-600 nm. When the light curing had been finished, the air valve 11 was opened to bring the gas pressure in the unit back to ambient pressure, and the light-cured resin denture base and stone cast were removed.

The denture base thus obtained was completely light-cured despite a short light-curing time and was very compatible with the stone cast.

Furthermore, a contact surface of EPOREX-D 6 with the stone cast 7, that is, the interface between a tissue side of the denture base and the stone cast 7, did not contain any air bubbles at all, and the contact surface looked very smooth and shiny.

COMPARATIVE EXPERIMENT 1

The resin sheet of EPOREX-D placed on the stone cast was light-cured for 10 minutes by using a visible light emitter LABOLIGHT LV-I (G-C DENTAL INDUSTRIAL CORP.) instead of U-LINE 2 without using the gas-pressure unit or the sheet.

Compatibility of the denture base thus obtained was inferior to that of EXAMPLE 1.

Furthermore, a few air bubbles were found in the interface between the denture base and the stone cast, which seemed to have been formed at the time when the resin sheet was pressed on the stone cast. The surface of the denture base looked rough.

EXAMPLE 2 - See Figure 1

At first, a light-curing resin sheet of TRIAD (Dentsply International Inc) 6 was placed on top of the stone cast 7 provided with clasps and labial guide wire which had been set at a predetermined position, and then set on the bed plate 12.

Then the flexible silicon rubber sheet 9 was placed on the sheet fastener 10 and fixed, and after the screw height adjuster 13 was adjusted to control the position of the stone cast 7, the first air valve 8 and the second air valve 11 were closed.

Thereafter, by opening the compressed gas valve 3, compressed air in the air compressor 1 was allowed to flow into the light-curing unit 5 which was provided with the quartz glass board 14 until the gas pressure in the unit reached 4 bar.

After closing the valve 3, the resin sheet 6 was light-cured for 3 minutes using 250 W Multi-mirror Project Lamp EXY (a halogen lamp manufactured by GENERAL ELECTRIC) 4 which emitted visible light with a wave length of 400-600 nm. When the light curing had been finished, the air-conducting bulb 11 was opened to bring the gas pressure in the unit back to ambient pressure, and the retaining appliance and the stone cast were removed.

The retaining appliance thus obtained was completely light-cured despite a short light-curing time and was very well compatible with the stone cast.

Furthermore, a contact surface of the TRIAD 6 with the stone cast 7, that is, the interface between the tissue side of the retaining appliance and the stone cast, did not contain any air bubbles at all, and details of the surface were very well reproduced. The contact surface looked very smooth and shiny.

COMPARATIVE EXPERIMENT 2

The resin sheet of TRIAD placed on the stone cast was light-cured for 10 minutes by using a visible light emitter TRIAD II (Dentsply International Inc) instead of Multi-mirror Project Lamp EXY without using the gas-pressure unit or the sheet.

Compatibility of the retaining appliance thus obtained was inferior to that of EXAMPLE 2.

Furthermore, many air bubbles were found at parts corresponding to rugae of palate and lingual cervical area in the interface between the retaining appliance and the stone cast, as well as around the lug of the clasps and labial guide wire. It seemed that these air bubbles had been formed due to insufficient adaptation of the resin sheet. The surface of the retaining appliance looked rough.

EXAMPLE 3 - See Figure 1.

At first, an amount of New METACOLOR resin paste (Sun Medical Co. Ltd.) was built up over a metal frame provided with a maxillary 3 - 3 bridge and supplied with details on its surface so as to complete the metal frame. The completed metal frame was then set on the bed plate 12, and the first air valve 8 and the

second air valve 11 were closed.

Thereafter, by opening the compressed gas valve 3, compressed nitrogen gas from a gas cylinder was allowed to flow into the light-curing unit 5 which was provided with the quartz glass board 14 until the gas pressure in the unit reached 8 bar.

5 After closing the valve 3, the resin sheet 6 was light-cured for 60 seconds using 75 W CERAMAX (a xenon lamp manufactured by ILC Technology) 4 which emitted visible light with a wave length of 400-600 nm. When the light curing had been finished, the air valve 8 was opened to bring the gas pressure in the unit back to ambient pressure, and the metal frame of the bridge was removed.

10 The bridge thus obtained was completely light-cured despite a short light-curing time. Since there was no unpolymerized layer on its surface, the surface could be glossy after only brief polishing without damaging the details on the surface.

Bubbles contained in the resin paste had disappeared due to the gas pressure during the light curing, resulting in translucent and aesthetic facing resins of the bridge. When adhered to the abutment, the resulting bridge could function very well because the metal frame was tightly adhered to the cured resins.

15

COMPARATIVE EXPERIMENT 3

20 The resin paste of New METACOLOR (Sun Medical Co. Ltd) was built up on the same metal frame as in EXAMPLE 3, and irradiated for two minutes by a visible light emitter Dentacolor XS (Kulzer GmbH) instead of CERAMAX without exerting gas pressure.

The bridge thus obtained had an unpolymerized layer on its surface, which required cumbersome grinding and polishing by using a bur in order to form the shape on the surface. Furthermore, small bubbles were found, which seemed to have been generated during the lamination of resin pastes of dentine color and enamel color. The surface finish thus obtained was much poorer than that obtain in EXAMPLE 3. After 25 the bridge had been used in the mouth for a long time, discoloration and cracks in the resin were found at the interface between the metal and the resin where it had been insufficiently adhered to the metal.

EXAMPLE 4 - See Figure 2

30 At first, the resin sheet of EPOREX-D (light-curing base resin by Nippon Oil and Fats Co. Ltd) 6 was placed on top of the stone cast 7 having the shape of an edentulous jaw, evenly finger-pressed and then set on the bed plate 12.

35 Then a flexible polyvinyl chloride sheet 9 with a thickness of 1.5 mm was placed on the sheet fastener 10, and after a height adjuster 16 was moved by using an elevator 18 in order to adjust the position of the stone cast, the air valve 11 was closed. An infrared-ray heater 15 was then used to heat the sheet at 200°C for 60 seconds to make it more flexible.

40 Thereafter, by opening the compressed gas valve 3, compressed air in the air compressor 1 was allowed to flow into a light-curing unit 17 which was provided with the quartz glass board 14 until the gas pressure in the unit reached 4 bar.

45 After closing the valve 3, the resin sheet 6 was light-cured for four minutes using lamp 4 (27 W U-LINE 2) which emitted visible light with a wave length of 400-600 nm. When the light curing had been finished, the air valve 11 was opened to bring the gas pressure in the unit back to ambient pressure, and the light-cured denture base and stone cast were removed.

The denture base thus obtained was completely light-cured despite a short light-curing time and was very compatible with the stone cast. There was found no deformation on the denture base.

50 Furthermore, a contact surface of EPOREX-D 6 with the stone cast 7, that is, the interface between the tissue side of the denture base and the stone cast, did not contain any air bubbles at all, and the contact surface looked very smooth and shiny.

COMPARATIVE EXPERIMENT 4

55 The resin sheet of EPOREX-D placed on the stone cast was light-cured for 10 minutes by using LABOLIGHT LV-I instead of U-LINE 2 without using the gas-pressure unit, the sheet nor the heating unit.

Compatibility of the denture base thus obtained was inferior to that of EXAMPLE 4.

Furthermore, a few air bubbles were found in the interface between the tissue side of the denture base

and the stone cast, which seemed to have been formed at the time when the resin sheet was pressed on the stone cast. The surface of the denture base looked rough.

5 EXAMPLE 5 - See Figure 3.

At first, the resin sheet of TRIAD (Dentsply International Inc) 6 was placed on top of the stone cast 7 provided with clasps 20 and labial guide wire which had been set at a predetermined position, and then set on the bed plate 12.

10 Then an ethylene-vinyl acetate copolymer sheet (9) 1 mm thick was placed on the sheet fastener 10, and after the height adjuster 16 was adjusted to control the position of the stone cast 7, the air valve 11 was closed.

Thereafter, by opening the compressed gas valve 3, compressed air heated to 120°C by an electrical heater 19 was allowed to flow from the air compressor 1 into the light-curing unit 17 which was provided 15 with the quartz glass board 14 until the gas pressure in the unit reached 3 bar.

After closing the valve 3, the resin sheet 6 was light-cured for two minutes using 250 W Multi-mirror Project Lamp EXY (a halogen lamp manufactured by GENERAL ELECTRIC) 4 which emitted visible light with a wave length of 400-600 nm. When the light curing had been finished, the air-conducting bulb 11 was 20 opened to bring the gas pressure in the unit back to ambient pressure, and the retaining appliance and stone cast were removed.

The retaining appliance thus obtained was completely light-cured despite a short light-curing time and had no deformation. It was very compatible with the stone cast.

Furthermore, the contact surface of TRIAD 6 with the stone cast 7, that is, the interface between the tissue side of the retaining appliance and the stone cast, did not contain any air bubbles at all, and details 25 of the surface were very well reproduced. The contact surface looked very smooth and shiny.

Since resin around the lug of the clasps and labial guide wire retained its strength and adhered well to the metal, after using in the mouth, no discoloration of the resin due to cracks or stains was found.

30 COMPARATIVE EXPERIMENT 5

The resin sheet of TRIAD placed on the stone cast was light-cured for 10 minutes by using TRIAD II instead of Multi-mirror Project Lamp EXY without using the gas-pressure unit, the sheet nor the heating unit.

The compatibility of the retaining appliance thus obtained was inferior to that of EXAMPLE 5.

35 Furthermore, many air bubbles were found at parts corresponding to rugae of palate and lingual cervical area in the interface between the tissue side of the retaining appliance and the stone cast, as well as around the lug of the clasps and labial guide wire. It seemed that these air bubbles had been formed due to insufficient pressure being exerted on the resin sheet. The surface of the retaining appliance looked rough.

40 After using in the mouth, discoloration due to cracking or deformation of the resin around the lug of the clasps and labial guide wire, and deformation in its tissue side.

EXAMPLE 6

45 Test samples prepared according to the above EXAMPLES and COMPARATIVE EXPERIMENTS were subjected to universal testing machine for determining bending strength thereof. The size of the test samples were 2 x 2 x 25 mm for EXAMPLES 1 to 3 and COMPARATIVE EXPERIMENTS 1 to 3, and 65 x 10 x 2.5 mm for EXAMPLES 4 and 5 and COMPARATIVE EXPERIMENTS 4 and 5.

The results obtained are summarized in the following TABLE.

50

55

Bending Strengths (MNm ⁻²)	Bending Strengths (MNm ⁻²)
EXAMPLE 1 123	COMPARATIVE 1 91
EXAMPLE 2 102	COMPARATIVE 2 79
EXAMPLE 3 108	COMPARATIVE 3 71
EXAMPLE 4 130	COMPARATIVE 4 91
EXAMPLE 5 119	COMPARATIVE 5 78

As seen from the results, the bending strengths of the cured resins prepared in EXAMPLES of the present invention are higher than those of the corresponding COMPARATIVE EXPERIMENTS.

Claims

1. A method for light curing of dental light-curing resins, characterised in that gas pressure above ambient pressure is exerted on the resin during the light-curing.

2. A method for light curing according to claim 1, characterised in that the surface of the dental light-curing resin is covered by a flexible sheet and that the gas pressure is exerted through the sheet, the sheet preferably being transparent or translucent.

3. A method for the light curing according to claim 2, characterised in that the dental light-curing resins are pressed and fixed on a support and that the sheet is heated before or during the light curing.

4. A method for the light curing according to claim 1, 2 or 3, characterised in that visible light is used for the light-curing.

5. A method for light curing according to any one of the preceding claims, characterised in that the gas pressure is in the range 2 to 7 bar.

6. An apparatus for light curing of resins comprising: a chamber (5), lighting means (4) and means (1,2,3,7,8,11) for selectively exerting a gas pressure above ambient pressure on the resin in the chamber.

7. An apparatus according to claim 6, characterised in that at least a part (14) of the apparatus is made of transparent material.

8. An apparatus according to claims 6 or 7, characterised by a flexible sheet (9) across which a pressure may be exerted, said sheet preferably being transparent or translucent.

9. An apparatus according to any one of claims 6, 7 or 8, characterised in that it comprises means (10) for fastening the edge of the transparent or translucent and flexible sheet (9)

10. An apparatus according to any one of claims 6, 7, 8 and 9, characterised in that it further has an adjustable support (12) for the light-curing resin and a heating unit.

11. An apparatus according to any one of claims 6, 7, 8, 9 or 10, characterised in that visible light is used for the light curing.

Fig. 1

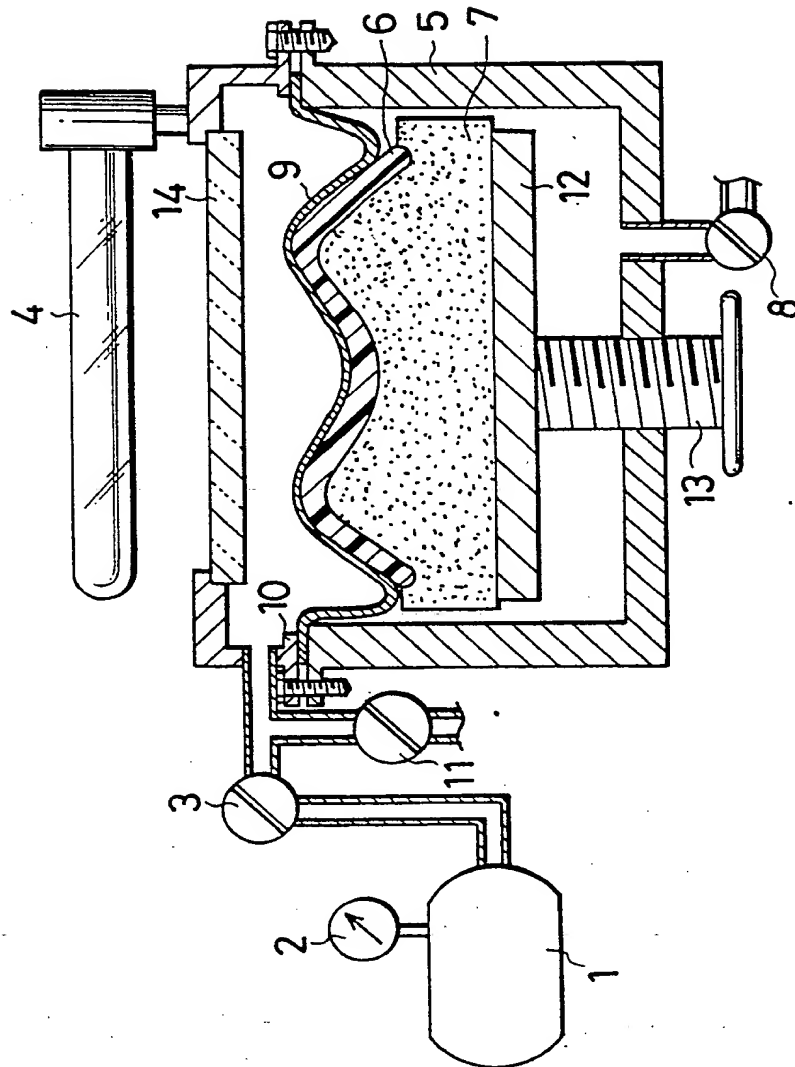


Fig. 2

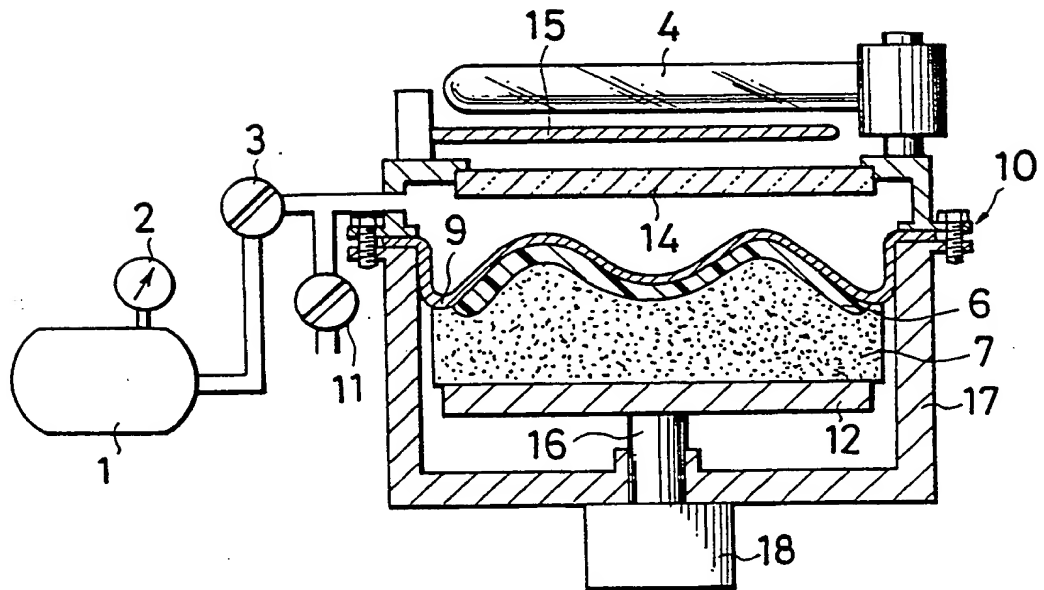
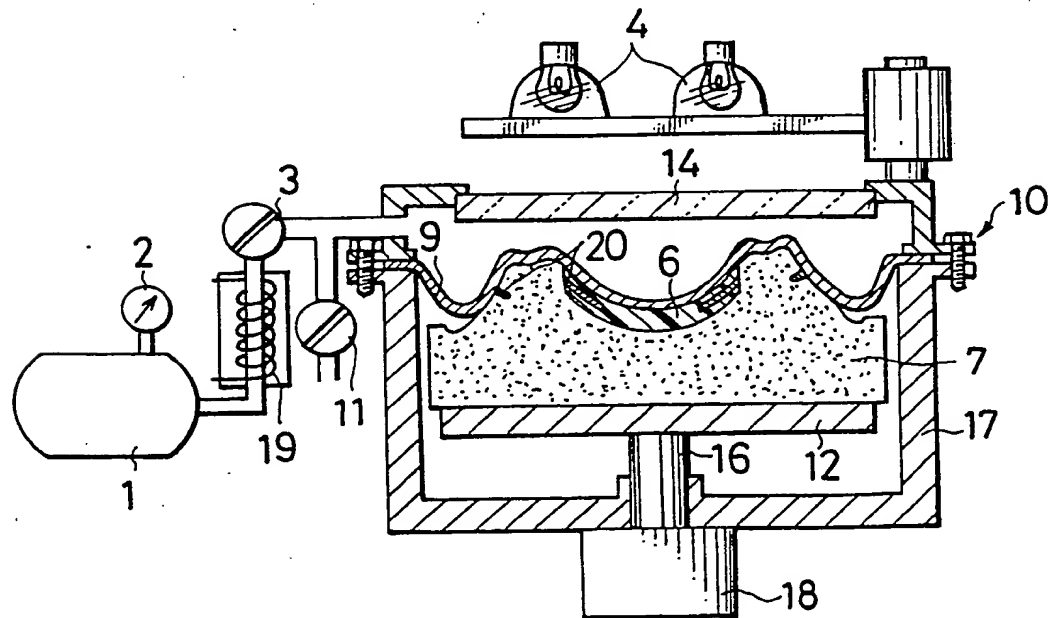


Fig. 3





DOCUMENTS CONSIDERED TO BE RELEVANT			EP 90300809.2
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int Cl')
A	<u>US - A - 4 645 649</u> (KUNIHICO NAGAO) * Abstract * --	1, 6	A 61 C 13/087 A 61 K 6/08
A	<u>DE - A1 - 3 637 371</u> (FRIEDRICH SCHILLER-UNIVERSI- TÄT-JENA) * Abstract * ----	1	
			TECHNICAL FIELDS SEARCHED (Int Cl')
			A 61 C A 61 K
The present search report has been drawn up for all claims			
Place of search VIENNA		Date of completion of the search 15-03-1990	Examiner HEIN
CATEGORY OF CITED DOCUMENTS			
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			
T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			